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(1*S*,3*R*,4*R*)-1,3,4,6-Tetra-*O*-acetyl-2-*O*-methylsulfonyl-α-D-mannopyranoside

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Key indicators

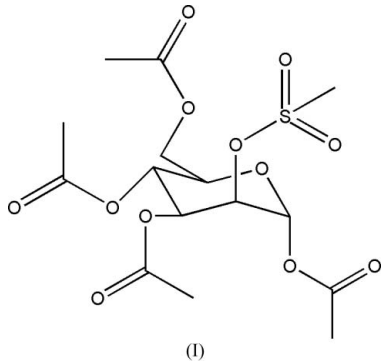
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.038
wR factor = 0.091
Data-to-parameter ratio = 16.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{15}H_{22}O_{12}S$, the pyranose ring adopts a chair conformation.

Comment

The title compound, (I), is a key intermediate in the synthesis of 2-deoxy-2-[18F]-fluoro-D-glucose ([18F]FDG; Hamacher *et al.*, 1986), a compound which measures glucose cellular uptake in the body and is one of the most widely used molecular imaging probes in positron emission tomography (PET).



(I)

In the crystal structure of the title compound, (I), the pyranose ring adopts a chair conformation (Fig. 1). All geometrical parameters are normal. The configurations of the chiral C atoms in the molecule are as follows: C1 *S*, C2 *S*, C3 *R*, C4 *R* and C5 *R*.

Experimental

A solution of 1,3,4,6-tetra-O-acetyl-α-D-mannopyranoside (0.6 g, 1.72 mmol) in dichloromethane (20 ml) was added dropwise over 20 min, with stirring, to methanesulfonyl chloride (0.4 ml, 5.17 mmol) and pyridine (10 ml) at 273 K. The mixture was allowed to warm to room temperature and was stirred overnight, after which water (20 ml) was added and the layers were separated. The aqueous phase was extracted with more dichloromethane, the combined organic layers were dried (Na_2SO_4) and evaporated, and the residue was chromatographed on silica with light petroleum–ethyl acetate (1:1 *v*/*v*) as eluent to give the product (yield 0.66 g, 88%). Single crystals of (I) (m.p. 383–385 K) were obtained by the slow evaporation of an ethyl acetate and petroleum ether (1:2 *v*/*v*) solution.

Crystal data

$C_{15}H_{22}O_{12}S$
M_r = 426.39
Orthorhombic, $P2_12_12_1$
a = 8.947 (2) Å
b = 10.373 (3) Å
c = 22.477 (6) Å
V = 2086.2 (9) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.21 mm^{-1}
T = 294 (2) K
0.28 × 0.24 × 0.18 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.943, T_{max} = 0.963
11993 measured reflections
4249 independent reflections
2968 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.091
S = 0.99
4249 reflections
257 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack (1983), 1794 Friedel pairs
Flack parameter: 0.06 (8)

The H atoms were positioned geometrically and refined in the riding-model approximation, with C—H = 0.96 (methyl), 0.97 (CH_2) and 0.98 Å (CH), and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

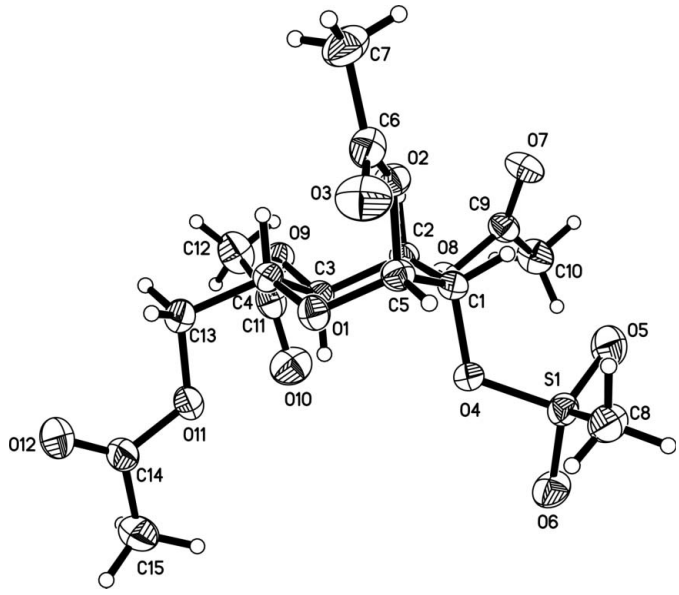


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.

References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 6.0) **[both Version 5.01??]** and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Hamacher, K., Coenen, H. H. & Stocklin, G. (1986). *J. Nucl. Med.* **27**, 235–238.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.